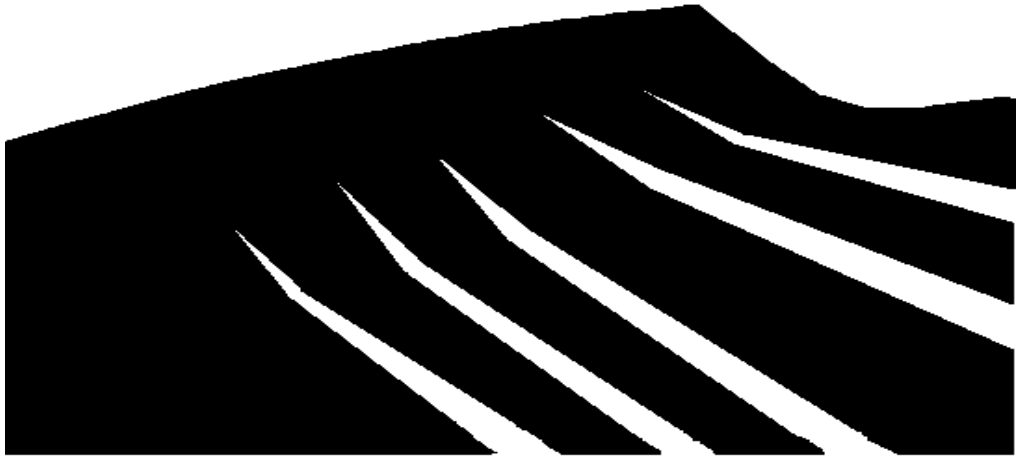
SORPTION AND DESORPTION RATIO DETERMINATIONS BY A BATCH SAMPLE TECHNIQUE FOR THE DYNAMIC TRANSPORT TASK

LOS ALAMOS
QUALITY PROGRAM



APPROVAL FOR RELEASE

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Los Alamos
Yucca Mountain Site Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	10/21/91	N/A	Initial procedure.
R1	08/08/94	All	Updated and revised procedure per QP-06.3.
R2	12/23/96	All	Revised procedure to comply with LANL-YMP-QP-06.3 requirements.

SORPTION AND DESORPTION RATIO DETERMINATIONS BY A BATCH SAMPLE TECHNIQUE FOR THE DYNAMIC TRANSPORT TASK

1.0 PURPOSE

The purpose of this procedure is to delineate the method for determining batch sorption and desorption coefficients for the distribution of various chemical species between geologic materials and natural or synthetic waters. The Dynamic Transport Task uses columns of different configurations to determine the characteristics of the geologic media.

2.0 SCOPE

This procedure defines guidelines to be used for sorption and desorption studies in the tasks of the Los Alamos National Laboratory (Los Alamos) Yucca Mountain Project (YMP).

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management
LANL-INC-DP-35, pH Measurements
LANL-CST-DP-63, Preparation of NTS Core Samples for Crushed Rock Experiments
LANL-CST-DP-99, Collection of Bulk Well and Spring Water Samples
CST Division Environmental Safety and Health Operational Statement

4.0 DEFINITIONS

4.1 The measured sorption or desorption ratio, Kd, (at equilibrium) is defined as:

$$Kd = \frac{\text{amount or activity of the sorbing or desorbing species per g of solid}}{\text{amount or activity of the sorbing or desorbing species per ml of solution}}$$

4.2 Tracer solution

Tracer solutions are comprised of either synthetic water or natural water from Yucca Mountain along with a specified concentration of the species whose sorption behavior is to be studies (for example, neptunium, plutonium or uranium).

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure:

- Principle Investigator (PI)
- Users of this Detailed Procedure (DP)

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this QP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, Section 6.1.6.
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 This procedure can be used to study sorption or desorption in the Dynamic Transport Task of the Los Alamos YMP. This procedure can be utilized to measure a sorption ratio. If sorption equilibrium is attained during the experiment, the measured ratio is the distribution coefficient.

6.2 Equipment and Hardware/Software

- Containers with a leak-proof cap (high temperature experiments require a screw cap sealing assembly with a rubber O’ring)
- Shaker (high temperature experiments require a high-temperature shaker referred to as a thermal block; See Attachment 1, Figure 1)
- Calibrated balances
- Analytical pipets
- Centrifuge capable of 15,000 (RCF) (Relative Centrifugal Force)

6.2.1 Equipment Malfunctions

There are no critical equipment malfunctions that are likely to occur that would interrupt this experiment.

6.2.2 Safety Considerations

The samples generated by this procedure can be radioactive; therefore, they should be handled in accordance with CST Division Environmental Safety and Health Operational Statement. The centrifuges must be operated in accordance with the manufacturer's instructions.

6.2.3 Special Handling

Before storing sample containers for analysis, seal the container with parafilm or curing silicon rubber sealant. Shipping is performed according to appropriate LANL procedure and samples are controlled pursuant to LANL-YMP-QP-08.1.

6.3 Preparatory Verification

Notebook Entries

The recording of data in the laboratory notebooks or binders can be performed by following the example spreadsheet templates on Attachment 2 and Attachment 3.

6.3.1 Hold Points

There are no hold points for this procedure.

6.3.2 Calibration

Balances used for weighing are required to be calibrated according to LANL-YMP-QP-12.3. When data are collected from a balance the unique identifier number of that balance is recorded in the user's laboratory notebook along with the data collected. pH meters are calibrated before measurements are taken. To ensure accuracy of sample pH measurements, buffers used for calibration should be measured prior to and after taking sample pH measurements. The unique identifier number of each pH meter used is recorded in the laboratory notebook or binder. (See Attachment 2).

6.3.3 Environmental Conditions

No special conditions are required for this DP. If any special conditions are utilized, they will be recorded in accordance with section 6.7 of this DP.

6.4 Control of Samples

All samples will be controlled using the following procedures:

- LANL-CST-DP-99, Collection of Bulk Well and Spring Water Samples
- TWS-INC-DP-63, Preparation of NTS Core Samples for Crushed Rock Experiments
- LANL-YMP-QP-08.1, Identification and Control of Samples

6.5 Implementing Procedure

6.5.1 Clean the containers to be used for the experiment. The containers are usually cleaned by rinsing with deionized water three times and drying without permitting contamination. Alternative cleaning routines will be documented in section 6.7 of this procedure.

6.5.2 Weigh an amount of the material to be used into a clean, weighed container (when running high temperature experiments, use containers with screw cap sealing assembly). Determine the weight of the container with the material to be used. Record the weight in the laboratory notebook or binder (see Attachment 3).

6.5.3 Add a known volume of the water to be used in the experiment to the container from step 6.5.2. Determine the weight of the container after addition of the water. Record the weight in the laboratory notebook or binder (see Attachment 3).

6.5.4 Shake the mixed phases (when running high temperature experiments, the container with the screw cap sealing assembly must be shaken in a high temperature shaker referred to as a thermal block). Normally the pre-equilibration step is performed for a period of 2-14 days.

6.5.5 Centrifuge the mixture for at least 2 hours at a minimum speed of 15,000 RCF.

6.5.6 Separate the phases (by decanting or pipetting the solution).

6.5.7 Determine the weight of the container after decantation. Record the weight in the laboratory notebook or binder (see Attachment 3).

6.5.8 Prepare the tracer solution to be used for this experiment and document the method used in the laboratory notebook.

6.5.9 Add an approximate volume of the tracer to be weighed to the preequilibrated material and record the julian time. Determine the

weight of the container, moist material and tracer solution added. Record the weight in the laboratory notebook or binder (see Attachment 3).

6.5.10 Take an aliquot of the tracer solution and analyze it using the appropriate analytical technique to determine the initial concentration of the tracer in the solution used. Take a second aliquot to determine the pH of the tracer solution following DP-35. Record the pH value in the laboratory notebook or binder (see Attachment 3).

6.5.11 Shake the container in item 6.5.9 (when running high temperature experiments, shake the container in the thermal block). Usually the container(s) is shaken for three weeks. If a different length of time is utilized, the change will follow section 6.7 of this DP.

6.5.12 After equilibration, weigh the container. Record the weight in the laboratory notebook or binder (see Attachment 3).

6.5.13 Separate the phases by filtration using a membrane-type filter of appropriate size or by centrifuging the sample(s) for one hour at a minimum of 15,000 RCF. After separation, take an aliquot of the filtrate (if filtration is used) or the supernatant (if centrifugation is used) and transfer to a clean container.

6.5.14 If desorption is going to be performed, remove the remaining supernatant, weigh the container with the moist material, and follow the desorption procedure. If the solid is going to be analyzed for determination of the amount of tracer in the solid phase, remove the remaining supernatant, take an aliquot of the moist material and weigh the aliquot. Let the aliquot dry, weigh the dried sample, and determine the amount of tracer in the solid using the appropriate analytical technique. Record the weight of the sample and the amount of tracer in the solid.

6.5.15 If separation is performed by filtration in step 6.5.13, use a portion of the filtrate for pH determination following DP-35. Record the pH value in the laboratory notebook or binder.

6.5.16 Continue the separation of the phases by centrifuging the aliquot taken in step 6.5.13 at a minimum of 15,000 RCF for one hour.

6.5.17 Take an aliquot of the centrifuged sample in step 6.5.16 and place in a clean container.

6.5.18 Continue the separation of the phases by centrifuging the aliquot taken in step 6.5.17 at a minimum of 15,000 RCF for two hours.

6.5.19 Take an aliquot of the centrifuged sample in step 6.5.18, place it in a clean container, and analyze it to determine the amount of tracer in the

solution using the appropriate analytical technique. The remaining solution is used for pH determination following DP-35.

6.6 Data Acquisition and Reduction

Calculate the Kd according to the definition given in section 4.0. This calculation can be performed directly if the amount of tracer in the solid phase has been measured. The calculation can also be performed by difference using the tracer concentrations in the initial and tracer-contacted solutions in order to determine the amount of tracer that abandoned the solution and is present in the solid phase.

6.7 Potential Sources of Error and Uncertainty

Sources of error and uncertainty include:

a) Incomplete separation of solid and liquid samples.

b) Leakage from the container during shaking or handling.

c) Improper recording or transfer of data.

d) If Kd values are out of range, the sample is rejected.

When work, as described in this DP, cannot be accomplished or would result in an undesirable situation, the work will be stopped and not resumed until this DP is revised to reflect the correct practice. However, if the revision of this DP cannot be accomplished in a timely manner, the continuation of the work will be described in accordance with LANL-YMP-QP-03.5.

The responsible PI or his/her designee will determine whether to use the collected data. If a decision to use the data is made, the justification for this decision must be entered in the investigator's logbook or binder.

7.0 RECORDS

Records resulting from the proper execution of this DP are entries in the laboratory notebooks and electronic media on which data is stored. These records are documented pursuant to LANL-YMP-QP-03.5.

All records should be submitted to the records processing center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

If non-optional data are recorded as designated in Attachments 2 and 3, and the PI accepts the experimental data, the data will be accepted as qualified for the YMP.

9.0 TRAINING

Staff members and technicians assigned to this work will be qualified by formal on-the job training under the supervision of qualified personnel according to LANL-YMP-QP-02.7.

10.0 ATTACHMENTS

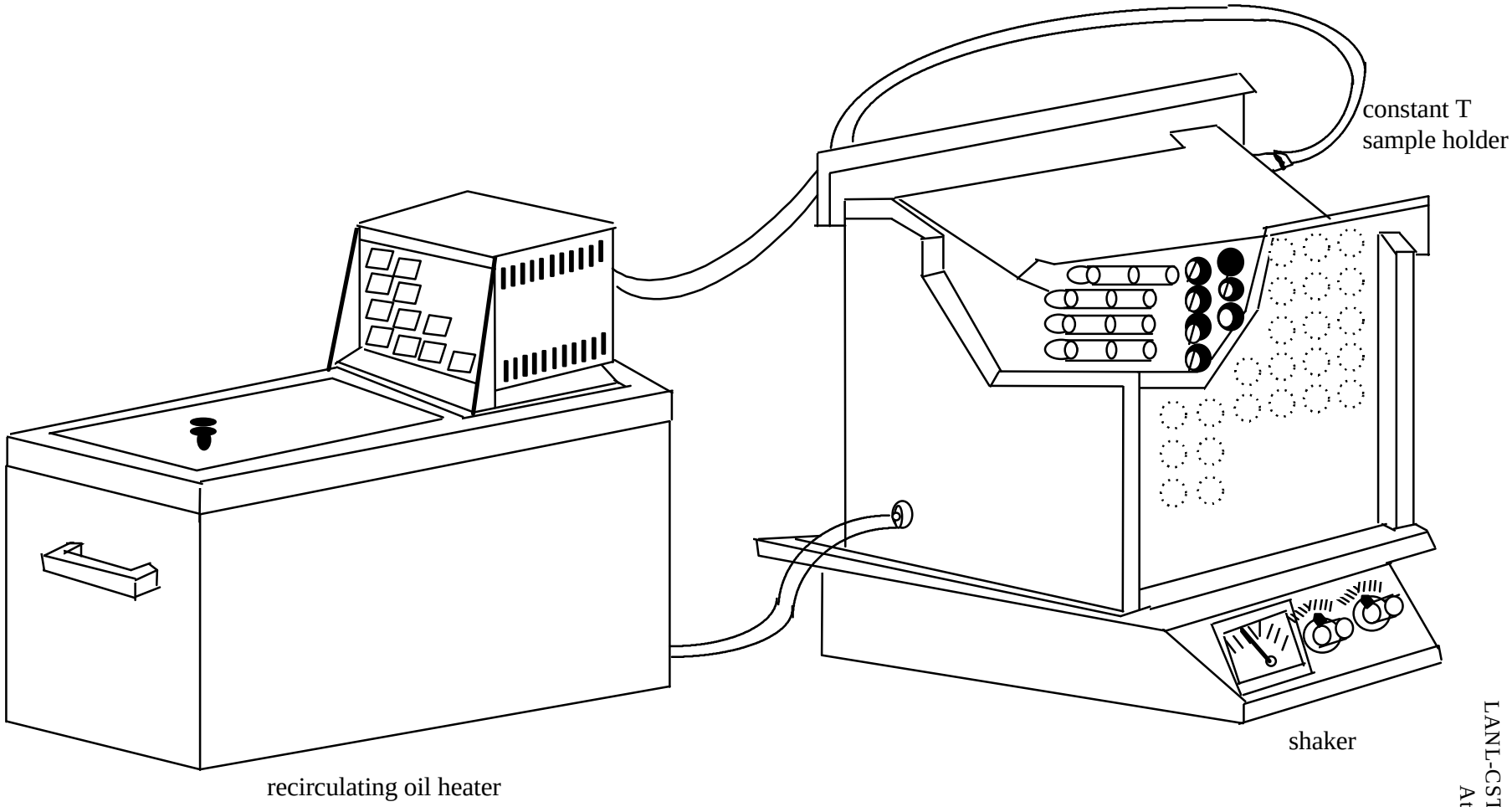
Attachments 2 and 3 are suggested formats; other formats may be used.

Attachment 1: Figure 1 (1 page)

Attachment 2: Data Sheet (3 pages)

Attachment 3: Spreadsheet (2 pages)

Figure 1: Batch Sorption Equipment for High T Experiments



YUCCA MOUNTAIN DATA INFORMATION SHEET	
Experiment Number	***required***
ID of water used in experiment	***required***
Binder Reference	***required***
Tracer ID	***required***
Binder with Information on Tracer Solution	***required***
Balance Used Before Addition of Radionuclide Tracer	***required***
Balance Used After Addition of Radionuclide Tracer	***required***
Mineral Code IDs	***required***
Sieving Information	***required***
Binder with Information on Solid Samples	***required***
Type of Oak Ridge Container Used for Sorption Experiment	***required***
Sorption Procedure Utilized	***required***
pH Measurement	
pH Procedure Used	***required***
pH meter	***required***
ID of pH Meter	***required***
Tracer Solution pH Before Adding to OR Tubes	***required***
Lot Number and Brand for pH 4 Buffer	
Reading for pH 4 Buffer (prior to making measurements)	
Reading for pH 4 Buffer (after making measurements)	
Lot Number and Brand for pH 7 Buffer	***required***
Reading for pH 7 Buffer (prior to making measurements)	***required***
Reading for pH 7 Buffer (after making measurements)	***required***
Lot Number and Brand for pH 10 Buffer	***required***
Reading for pH 10 Buffer (prior to making measurements)	***required***
Reading for pH 10 Buffer (after making measurements)	***required***
Eh Measurement	
Eh Procedure Used	TWS-INC-03-93-01,p. A12-A16
ID of Eh Meter	***required when pertinent***
Electrodes Used for Eh Measurement	***required when pertinent***
Zobell Lot Number and Ideal Value	***required when pertinent***
Zobell Solution preparation date	***required when pertinent***
Eh of the Zobell Solution (prior to making Measurements), mV	***required when pertinent***

Eh of the Zobell Solution (after making Measurements), mV	***required when pertinent***
Temperature Range for Eh Measurements, °C	***required when pertinent***
Eh of Water Used in the Experiment, mV	***required when pertinent***
Verification of Liquid Scintillation Counting	
ID of Liquid Scintillation Counter	***required when pertinent***
ID of Standard Counted	***required when pertinent***
Ideal Counts per Minute of Standard Counted	***required when pertinent***
Actual Counts per Minute	***required when pertinent***
ID of Standard Counted	***required when pertinent***
Ideal Counts per Minute of Standard Counted	***required when pertinent***
Actual Counts per Minute	***required when pertinent***
Procedure Utilized	***required when pertinent***
Vendor Sample Analysis Information	***required when pertinent***
Vendor Analysis Notebook Reference:	***required when pertinent***
Location of Data	***required***
Experimental Data (notebook #)	***required***
Experimental Data (page(s)#)	***required***
Reduced Data (optical disk#)	***required***
Reduced Data (subdirectory #)	***required***
Reduce Data (file Name)	***required***

Sample Identification	Experiment No.	Atmosphere	Desired Radionuclide Tracer Solution Concentration CPM/ml or ppm	Description of Solid	Particle Size, micrometers (if sieved)	Sieving Information	Type of Water Used if Wet Sieved
Required	*Required*				*Required*	*Required*	*Required*
Tube + Cap Mass, g	Tube + Cap + Mineral Mass, g	Mineral Mass, g	Tube + Cap + Mineral + Water Mass, g	Water Mass, g	Julian Date Pretreatment Started “94	Julian Date Pretreatment Ended “94	Period of Pretreatment, Days
Required	*Required*		*Required*		*Required*	*Required*	*Required*
Tube + Cap + Mineral + Water (after Pretreatment) Mass g	% Evaporation during Pretreatment	Tube + Cap + Mineral + Water (after Decantation Mass, g	pH of Water after Pretreatment	Eh of Water after Pretreatment, mV	Julian Date Tracer Solution was added, “94	Mass after Tracer Solution was added, g	Julian Date Sorption Started “94
Required		*Required*	*Required*		*Required*	*Required*	*Required*
Julian Date Sorption Ended “94	Period of Sorption, Days	Mass after Sorption Ended, g	% Evaporation during Sorption	Tracer Solution pH After Sorption (Recorded Inside Glovebox Before Spin-down)	Mass after Sorption and Decantation, g	pH of Tracer Solution after Sorption	Eh of Tracer Solution after Sorption
Required	*Required*	*Required*		*Required*		*Required*	

Mass of Counting Vial, g	Mass of Counting Vial + Aliquot of Tracer Solution (after Soprtion), g	Mass of Counting Vial + Aliquot of Tracer Solution (after Sorption) + Scintillation Gel, g	Mass of Counted Tracer Solution (after Sorption), g	Total CPM in Counted Tracer Solution (after Sorption)	CPM/g or ppm in Counted Tracer Solution (after Sorption)	Control Samples Identifications	Control Tube + Cap Mass, g
Required	*Required*			*Required*		*Required*	*Required*
Mass after Tracer Solution was added to Control tube, g	Mass after Sorption Ended in Control Tube, g	% Evaporation during Sorption in Control Tube	pH of Tracer Solution (after Sorption) in Control Tube	Eh of Tracer Solution (after Sorption) in Control Tube	Mass of Counting Vial, g	Mass of Counting Vial + Aliquot of Tracer Solution from Control Tube, g	Mass of Counting Vial + Aliquot of Tracer Solution from Control Tube + Scintillation Gel, g
Required	*Required*		*Required*		*Required*	*Required*	
Mass of Counted Tracer Solution from Control Tube, g	Total CPM in Control Tracer Solution Counted	CPM/g or ppm in Control Tracer Solution Counted	Tracer Solution (Used for Sorption) Sample Identifications	Mass of Counting Vial, g	Mass of Counting Vial + Aliquot of Tracer Solution (Used for Sorption), g	Mass of Counting Vial + Aliquot of Tracer Solution (Used for Sorption) + Scintillation Gel, g	Mass of Counted Tracer Solution (Used for Sorption), g
	Required		*Required*	*Required*	*Required*		
Total CPM in Counted Tracer Solution (Used for Sorption)	CPM/g or ppm in Counted Tracer Solution (Used for Sorption)	Water ID	Type of Scintillation Cocktail Used	ID of Tracer Solution Used for Sorption Experiments			
Required		*Required*		*Required*			